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I, JULIE BILLINGSLEY, TEAM LEADER EXAMINATION SUPPORT AND SALES hereby certify that annexed is a true copy of the Provisional specification in connection with Application No. 2003905881 for a patent by NATIVE FIRE PTY LIMITED as filed on 24 October 2003.

WITNESS my hand this
Third day of November 2004

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AUSTRALIA

Patents Act 1990

# PROVISIONAL SPECIFICATION

**Applicant(s):**

NATIVE FIRE PTY LIMITED

**Invention Title:**

EXTRACTION PROCESS

The invention is described in the following statement:

# EXTRACTION PROCESS

## FIELD OF THE INVENTION

The present invention relates to a method for extracting compounds from plant material. The invention further relates to spray formulations containing compounds extracted by the method.

## BACKGROUND TO THE INVENTION

Pesticidally active oils are used to control pests on various surfaces, particularly insect pests on plants.

In the agricultural and horticultural field, pesticidally active oils are commonly applied to plants by means of a formulation known as a "spray formulation". Spray formulations are formulations comprising a high quantity of a pesticidally active oil, and are capable of being applied to surfaces by spraying the formulation onto the surface. Such formulations are commonly sprayed onto a surface by mixing the formulation with water to form an emulsion and spraying the resultant emulsion onto the surface. Spray formulations typically comprise about 80% to 90% by weight of one or more pesticidally active oil(s) and about 10% to 20% by weight of one or more surfactant(s). The spray formulation may also contain a small amount, for example up to about 10% by weight, of other components. The pesticidally active oil may be of mineral (petroleum) origin, or may be of vegetable or animal origin. Pesticidally active oils commonly used in spray formulations include paraffinic oils in the range C12 to C28.

Many compounds extracted from plant material have pesticidal activity, such as insecticidal, fungicidal and/or antibacterial activity. For example, extracts from the plant *Tasmannia stipitata*, containing various extracted compounds, have insecticidal activity, and can be used as an insecticide for the control of phytophagous mites such as Two Spotted Mites (*Tetranychus urticae),* Greenhouse Thrips *(Thrips tabaci)* and Western Flower Thrips *(Frankliniella occidentalis).* It is believed that one of the insecticidally active compounds in the extracts from *Tasmannia stipitata* is the compound polygodial, a sesquiterpene dialdehyde.

The two main processes used for extracting compounds from plant material are steam distillation and solvent extraction.

Steam distillation typically involves passing steam through plant material and then condensing the steam. Alternatively, steam distillation may involve immersing the plant material in boiling water, boiling the mixture and condensing the steam. In either case, volatile compounds are extracted from the plant material and are condensed with the steam. Typically the extracted compounds are in the form of an oil that is insoluble in the condensed water, and which can be separated from the water by a simple decanting process.

Solvent extraction typically involves immersing the plant material in a solvent for a period of time and under conditions suitable for compounds to be extracted from the plant material into the solvent, and then physically separating the solvent from the plant material. The compounds extracted into the solvent are typically then separated from the solvent by heating the solvent containing the extracted compounds to evaporate the solvent, leaving a residue comprising extracted compounds. Solvents used in solvent extraction include alcohols, particularly methanol and ethanol, hydrocarbons, particularly hexane, ketones, particularly acetone, halogenated hydrocarbons, and ethers.

Prior art steam distillation and solvent extraction processes for extracting compounds from plant material often involve heating the plant material and/or the solvent containing the extracted compounds at some stage during the extraction process. Steam distillation involves exposing the plant material to elevated temperatures during the extraction process. Prior art solvent extraction processes using commonly used solvents, such as ethanol, typically include the step of heating the solvent containing the extracted compounds to separate the solvent from the extracted compounds. Solvent extraction may also involve heating the plant material with the solvent to enhance the rate of extraction of compounds from the plant material into the solvent. Some compounds in plant material, including many pesticidally active compounds in plant material, are heat sensitive and are destroyed or rendered inactive by heat, or when heated readily react with other compounds. Processes for extracting compounds from plant material which involve exposing the plant material or the extracted compounds to elevated temperatures are therefore not suitable for extracting heat sensitive compounds from plant material or result in low yields of the heat sensitive compounds. For example, polygodial, a compound that can be extracted from plants such as *Tasmannia stipitata*, is transformed at elevated temperatures into less active or inactive isomers, or reacts with other compounds at elevated temperatures.

Previously, heat sensitive compounds have been extracted from plant material using solvent extraction with solvents having a low boiling point, such as fluorocarbons. Whilst temperature-related problems during the extraction or solvent recovery process are avoided or minimized using such solvents, fluorocarbons have a number of environmental disadvantages. Potentially the most effective fluorocarbon solvents, the hydrochlorofluorocarbons, are covered by ozone protection legislation, which prescribes a well advanced phase-out schedule. The hydrofluorocarbons, whilst not implicated in ozone depletion, are powerful greenhouse gases and are less suitable for use as solvents in solvent extractions from plant material due to their generally poor solvency power. Fluoroethers can possibly be used as solvents to extract compounds, but their cost is likely to be prohibitive in commercial application.

It would be desirable to include pesticidally active compounds extracted from plant material in spray formulations to supplement the pesticidal activity of the pesticidally active oil in the formulation. However, many pesticidally active compounds in plant material are heat sensitive and are therefore difficult to extract from plant material using conventional steam or solvent extraction processes. Further, extracts from plant material obtained using steam distillation or solvent extraction processes have often proven very difficult to disperse or dissolve in pesticidally active oils such as paraffinic oils to form a stable spray formulation comprising the extracted compounds, that is, a spray formulation that does not separate into separate layers after a few hours. This is the case with extracts obtained from *Tasmannia stipitata* using prior art solvent extraction processes.

## SUMMARY OF THE INVENTION

The present inventors have surprisingly found that chemically modified oils, surfactants, mixtures thereof, and mixtures thereof with an oil, can be used as a solvent to extract compounds from plant material. The present inventors have further found that when a chemically modified oil, a surfactant, a mixture thereof or a mixture thereof with an oil is used as a solvent to extract compounds from plant material, the solvent containing the extracted compounds can be mixed with paraffinic oils of the type conventionally used in spray formulations to produce a spray formulation containing the extracted compounds, without requiring the step of separating the extracted compound from the solvent. In some embodiments, the solvent containing the extracted compounds is itself a spray formulation containing compounds extracted from the plant material.

In a first aspect, the present invention provides a method for extracting compounds from plant material, the method comprising the step of contacting the plant material with a solvent, wherein the solvent is selected from the group consisting of chemically modified oils; surfactants, mixtures thereof, and mixtures of one or more chemically modified oils and/or one or more surfactants with an oil that is not a chemically modified oil, under conditions effective to extract compounds from the plant material into the solvent.

In some embodiments, the solvent consists of one or more chemically modified oils, one or more surfactants, or a mixture of one or more chemically modified oils and one or more surfactants. In other embodiments of the invention, the solvent consists of one or more chemically modified oils, one or more surfactants, or a mixture thereof, in admixture with an oil that is not a chemically modified oil. The oil may be a mineral oil, a vegetable oil or an animal oil, and may be a pesticidally active oil such as a paraffinic oil. In such a case, the one or more chemically modified oils, the one or more surfactants or the mixture thereof, may for example constitute from 5% to 99% by weight of the solvent, eg, from 5 to 10%, from 5 to 20%, more than 20%, more than 40%, more than 50% or more than 75% by weight of the solvent. If at least 70% of the solvent is a pesticidally active oil, then the resultant solvent containing the extracted compounds is a spray formulation and may be used without further modification to control pests.

The chemically modified oil may be any chemically modified oil. Preferably, the chemically modified oil is an esterified oil. The chemically modified oil may be a pesticidally active oil.

The surfactant may be any surfactant. Preferably the surfactant is a non-ionic surfactant.

If the method is to be used to directly produce a spray formulation containing compounds extracted from the plant material (i.e. to produce such a spray formulation without requiring the further step of mixing the solvent containing the extracted compounds with a pesticidally active oil), the solvent preferably comprises at least 70% by weight of a pesticidally active oil. An advantage of using such a solvent is that the resultant solvent containing the compounds extracted from the plant material can be used as a spray formulation without the need to first separate the extracted compounds from the solvent and subsequently mix the extracted compounds with a pesticidally active oil. This provides a significant advantage over prior art methods of extracting

compounds from plant material for use in spray formulations, resulting in substantial cost and time savings.

If the solvent containing the extracted compounds is to be mixed with a pesticidally active oil, such as a paraffinic oil, to form a spray formulation, the solvent is preferably miscible with, or soluble in, the pesticidally active oil. An advantage of using such a solvent is that the solvent containing the extracted compounds can be mixed with the pesticidally active oil to produce a spray formulation containing the compounds extracted from the plant material, without the need to first separate the extracted compounds from the solvent and then mix the extracted compounds with the pesticidally active oil. This provides a significant advantage over prior art methods of extracting compounds from plant material for use in spray formulations, resulting in substantial cost and time savings. Further, the present inventors have found that by using such a solvent, compounds can be extracted from the plant material into the solvent, and the solvent containing the extracted compounds can be mixed with the pesticidally active oil, to form a stable spray formulation comprising the extracted compounds.

The method of the first aspect of the present invention can be carried out at room temperature. Thus the method can be carried out to produce a solvent containing compounds extracted from the plant material, and the resultant solvent containing the extracted compounds can be used as, or incorporated into, a spray formulation, without exposing the plant material or the compounds extracted from the plant material to above room temperatures, either during the extraction process or during a subsequent step of separating the solvent from the extracted compounds. Thus, heat sensitive compounds can be extracted from the plant material and incorporated into a spray formulation.

In a second aspect, the present invention provides a solvent containing compounds extracted from plant material, wherein the solvent containing compounds extracted from plant material is produced by the method of the first aspect of the present invention.

In a third aspect, the present invention provides a spray formulation comprising a solvent containing compounds extracted from plant material, wherein the solvent containing compounds extracted from plant material is produced by the method of the first aspect of the present invention.

In a fourth aspect, the present invention provides a method for producing a spray

formulation containing compounds extracted from plant material, the method comprising the steps of:

(a) contacting plant material with a solvent, wherein the solvent is selected from the group consisting of chemically modified oils, surfactants, mixtures thereof, and mixtures of one or more chemically modified oils and/or one or more surfactants with an oil that is not a chemically modified oil, under conditions effective to extract compounds from the plant material into the solvent;

(b) separating the solvent from the plant material, for example by filtration; and

(c) mixing the solvent with a pesticidally active oil to produce a spray formulation containing compounds extracted from the plant material.

Typically the spray formulation produced by the method of the fourth aspect of the present invention comprises more than 70%, preferably about 80 to 90%, by weight of one or more pesticidally active oils.

In a fifth aspect, the present invention provides a spray formulation produced by the method of the fourth aspect of the present invention.

In a sixth aspect, the present invention provides a method for repelling insects from a surface, killing insects on a surface or inhibiting egg laying by insects on a surface, the method comprising the step of applying to the surface a spray formulation according to the third aspect of the present invention or a spray formulation according to the fifth aspect of the present invention.

BEST MODE OF PERFORMING THE INVENTION

As used herein, the term "chemically modified oil" refers to a compound or mixture of compounds prepared by chemically modifying a mineral, vegetable or animal oil, and which on contact with plant material is capable of extracting compounds from plant material into the chemically modified oil. Common ways of chemically modifying oils are by esterification, by hydrogenation or by sulphonation reactions. An example of a chemically modified oil is esterified vegetable oil. A chemically modified oil may or may not itself be an oil.

As used herein, the term "vegetable oil" refers to an oil derived from a plant material without chemically modifying the oil present in the plant material. For example, the

vegetable oil may be derived from plant material by pressing the plant material to extract the oil. As used herein, the term "animal oil" refers to an oil of animal origin derived from the animal without chemically modifying the oil.

As used herein, the term "pesticidally active compound" refers to a compound that repels, kills or otherwise adversely affects arthropod pests that cause physical damage to plants and/or transfer microorganisms that cause fungal or bacterial diseases to plants, and/or repels, kills or otherwise adversely affects microorganisms that cause fungal or bacterial diseases in plants. Pesticidally active compounds that can be extracted from plant material include, for example, citral, polygodial, anethole, azadirachtin, citronellal, alpha pinene, beta pinene, caryophyllene, guaiol, linalool, pyrethrin, quinine, terpineol and vanillin.

As used herein, the term "pesticidally active oil" refers to an oil that repels, kills or otherwise adversely affects arthropod pests that cause physical damage to plants and/or transfer microorganisms that cause fungal or bacterial diseases to plants, and/or repels, kills or otherwise adversely affects microorganisms that cause fungal or bacterial diseases in plants.

**Solvent**

The methods of the first and fourth aspects of the present invention comprise the step of contacting plant material with a solvent selected from one or more chemically modified oil(s), one or more surfactant(s), a mixture of one or more chemically modified oil(s) and one or more surfactant(s), or a mixture of one or more chemically modified oil(s) and/or one or more surfactant(s) with an oil that is not a chemically modified oil.

The chemically modified oil may be a chemically modified vegetable oil, a chemically modified mineral (petroleum) oil or a chemically modified animal oil.

Preferably, the chemically modified oil is an esterified oil, preferably an esterified vegetable or animal oil. The present inventors have found that esterified oils typically have a higher solvency power than unmodified vegetable, animal or mineral oils.

More preferably, the chemically modified oil is an esterified vegetable oil. Vegetable oils contain fatty acids which are present as triglycerides. Esterified vegetable oils can be produced by reacting these triglycerides with an alcohol such as methanol or ethanol in the presence of an alkaline catalyst. This reaction produces a mixture of fatty acid

esters and glycerol. The glycerol is then separated leaving fatty acid esters (an esterified vegetable oil).

For example, canola oil is rich in oleic acid, generally containing greater than 60% and often 80% by weight oleic acid. This oleic acid, together with other fatty acids such as linolenic acid, is present in canola oil in the form of triglycerides. When the oil is reacted with ethanol in the presence of an alkaline catalyst at about 50°C, a mixture of glycerol and fatty acid esters is formed. The glycerol is then removed, leaving a mixture rich in the ethyl ester of oleic acid. A similar reaction with methanol yields a mixture rich in the methyl ester of oleic acid. The methyl and ethyl esters of oleic acid are examples of esterified vegetable oils, and can be used as the solvent in the method of the first or fourth aspect of the present invention.

Similar esterification reactions can be carried out with vegetable oil obtained from soybean, sunflower, safflower, canola, corn, cotton, coconut, cocoa, castor or olive, to produce esterified vegetable oils suitable for use in the present invention.

Esterified vegetable oils include methyl, ethyl, propyl, and butyl esters of vegetable oils, or combinations thereof.

Examples of esterified vegetable oils which may be obtained from vegetable oils include:

Oleic acid, methyl ester;
Oleic acid, ethyl ester; and
Octadecanoic acid, butyl ester.

The chemically modified oil may also be a surfactant.

In some embodiments of the invention, the solvent is a surfactant, a mixture of one or more chemically modified oils and one or more surfactants, a mixture of one or more surfactants and an oil that is not a chemically modified oil, or a mixture of one or more surfactants and one or more chemically modified oils and an oil that is not a chemically modified oil.

The surfactant may be an anionic, cationic or non-ionic surfactant. If the solvent containing the extracted compounds is to be used as a spray formulation, or is to be mixed with a pesticidally active oil to produce a spray formulation, it is preferred that the surfactant is a non-ionic surfactant.

Examples of non-ionic surfactants that can be used in the method of the present invention include:

Glycerol esters of fatty acids
Polyethylene glycol dioleate
9-Octadecenoic acid monoester with 1,2-propanediol
Polyethylene glycol, dioleate
Polyethylene glycol, monococonut ester
Polyethylene glycol, monooleate
Diethylene glycol, monooleate

It is preferred that the solvent consist of or include one or more chemically modified oils that is not a surfactant. This is because surfactants generally have a relatively high viscosity and if the solvent is a surfactant or mixture of surfactants, the high viscosity of the surfactant(s) can create difficulties during the extraction process. For example, the high viscosity can create complications in the separation (e.g. filtration) of the plant material from the solvent after the solvent has been contacted with the plant material. In addition, if the solvent containing the compounds extracted from the plant material is to be used as or in a spray formulation, it is preferred that the solvent consist of or include one or more chemically modified oils that is not a surfactant as surfactants can cause phytotoxicity in plants to which the surfactant is applied, and thus high levels of surfactants in a spray formulation can cause phytotoxicity problems when the formulation is applied to certain plants. However, these problem may be overcome or minimised if the surfactant is used in combination with a chemically modified oil that is not itself a surfactant.

In some embodiments of the method of the first or fourth aspect of the present invention, a second solvent such as ethanol, acetone, glycerol or hexane may be mixed with the solvent to enhance the solvency power of the solvent. Such a second solvent may comprise from about 5% to about 50%, for example from about 5% to about 20%, by weight of the total weight of the solvent and the second solvent used in the extraction process.

Preferably the second solvent is selected such that it does not need to be separated from the extracted compounds prior to use of the extracted compounds, for example, the blending of the solvent containing the extracted compounds with a pesticidally active oil to form a spray formulation.

**Plant material**

The present invention can be used to extract compounds from a wide variety of plant material. The extracted compounds may have a variety of uses, including as a pharmaceutical, a colouring agent, a flavouring, a fragrance, or a pesticide.

Pesticidally active compounds can be extracted from a wide variety of plants, including the following Australian native plants:

- Plants of the genus *Callitris,* in particular *Callitris glaucophylla* and *Callitris endlicheri*
- Plants of the genus *Tasmannia,* in particular *Tasmannia stipitata* and *Tasmannia lanceolata*
- Plants of the genus *Leptospermum,* in particular *Leptospermum polygalifolium, Leptospermum petersonii, Leptospermum grandiflorum, Leptospermum neglectum, Leptospermum speciosum, Leptospermum brevipes, Leptospermum oreophiilum* and *Leptospermum gregarium*
- Plants of the genus *Prostanthera,* in particular *Prostanthera incisa* and *Prostanthera rotundifolia*
- Plants of the genus *Rhodamnia,* in particular *Rhodamnia whiteana* and *Rhodamnia argentea*

- Plants of the genus *Melaleuca,* in particular *Melaleuca uncinta, Melaleuca stypheloides, Melaleuca quinquenervia* and *Melaleuca alternifolia*
- Plants of the genus *Phebalium*, in particular *Phebalium squameum* and *Phebalium dentatum*
- Plants of the genus *Eucalyptus*, in particular *Eucalyptus melanophloia* and *Eucalyptus cloeziana*
- Plants of the genus *Acacia*, in particular *Acacia howittii*
- Other plants including *Cryptocaria cunninghamii, Austromyrtus dulcis, Backhousia citriodora* and *Backhoiusia anisata* (also known as *Anetholea anisata)*

Pesticidally active compounds can, for example, also be extracted from the following plants not native to Australia:

- *Polygonum hydropiper*
- *Azadirachta indica* (neem)
- *Chrysanthemum cinerariaefolium* (pyrethrum)
- *Ginkgo biloba*
- *Nicotiana tabacum* (tobacco)
- *Derris elliptica*
- *Melia azadirachta*
- *Warburgia stuhlmannii*
- *Warburgia ugandensis*
- *Cannella winterana*
- *Drimys winteri*
- *Ailanthus altissima*
- *Glycosmis species*
- *Anabasis aphylla*
- *Ryania speciosa*

The plant material may comprise the whole or any part of a plant, including leaves, flowers, trunks, butts and roots.

**Extracting compounds from plant material**

A typical solvent for use in the method of the first aspect of the present invention where the resultant solvent containing the extracted compounds is intended to be used as or in a spray formulation, or for use in the method of the fourth aspect of the present invention, is a mixture of one or more esterified oil(s) that is not a surfactant and one or more surfactant(s) in the following proportions:

Esterified oil(s): 70-90% by weight

Surfactant(s): 10-30% by weight

Depending on the esterified oil and surfactant used, the plant material and the intended use of the solvent containing the extracted compounds, these ranges can however be broader.

Some commercially available mixtures of an esterified oil and surfactants can be used. One such product is sold under the brand name Hasten™, by Victorian Chemicals Pty Ltd, 37- 49 Appleton Street, Richmond VIC 3121, Australia. This product comprises ethylated canola oil blended with non-ionic surfactants.

Typically the plant material is pre-treated so that it is in an appropriate physical form to facilitate the extraction of the compounds. Typically this comprises treating the plant material to increase the surface area of the plant material, so that contact between the plant material and the solvent is increased. Commonly, some form of comminution process is used to reduce the particle size of the plant material. A particle size with a maximum dimension of 1-3 mm is normally adequate to achieve a good yield.

In some cases, the moisture content of the plant material is also reduced prior to contacting the plant material with the solvent. The reduction in moisture content should be carried out in a manner which minimises the loss of any volatile compounds desired to be extracted from the plant material, and minimises the destruction or inactivation of compounds desired to be extracted from the plant material.

Typically, the plant material is contacted with the solvent by passing the solvent past the plant material, or immersing the plant material in the solvent.

The extraction process may for example be carried out by the following procedure:

1. The solvent is placed in a vessel, preferably a vessel equipped with a high shear mixer. Where high shear agitation is used, it may not be necessary to reduce the particle size of the plant material prior to contacting the plant material with the solvent as this may occur during the mixing of the plant material and solvent.
2. Agitation of the solvent is commenced and the plant material is added progressively.
3. Optionally, if the compounds of interest are not heat sensitive, the mixture may be heated to enhance extraction rate and yield.
4. Agitation is continued until the plant material is dispersed and the extraction process is proceeding. Alternatively, agitation can be continued throughout the extraction process.
5. When a suitable amount of compounds have been extracted, the mixture is removed from the vessel and filtered or centrifuged to separate the solvent containing the extracted compounds from the plant material.
6. Additional extract may be obtained by subjecting the residue of plant material to pressure.
7. Beneficiation processes may be performed on the solvent containing the extracted compounds as necessary. For example, additional filtration steps can be performed, any moisture present in the solvent can be removed and/or the solvent can be passed through charcoal or activated clay to remove any colouring matter. Beneficiation can also involve the addition of other compounds, such as quinic, acetic or citric acid, to improve the stability of, and enhance the efficacy of, the extracted compounds (such as polygodial), or the addition of antioxidants such as tocopherols to further enhance stability and product shelf life.

The above process can, for example, be used to extract the compound citral from leaves of *Backhousia citriodora* (lemon myrtle) which have been air dried and milled to a

particle size of 2 mm, using a solvent consisting of a mixture of an esterified vegetable oil and non-ionic surfactants. Citral is known to possess useful fungicidal properties.

If the solvent used comprises 70 to 80% or more by weight of a pesticidally active oil, the solvent containing the extracted citral, when filtered to remove residual plant material, is itself suitable without further processing for use as a fungicidal spray formulation. If other solvents are used, the solvent containing the extracted citral may be mixed with one or more pesticidally active oils and optionally one or more surfactants to form a spray formulation.

In an alternative extraction process, the plant material may be contacted with the solvent by placing the plant material in contact with the solvent, and leaving the plant material in contact with the solvent for a few days (for example 2 to 4 days) to several weeks typically at room temperature. The amount of time the plant material is left in contact with the solvent will vary depending upon the particle size of the plant material, the temperature, the solvency power of the solvent and the desired yield of the extracted compounds.

The methods of the first and fourth aspects of the present invention are typically carried out at room temperatures (for example at about 10°C to about 30°C). However, if the compounds to be extracted are not heat sensitive, the methods can be carried out at higher temperatures.

The method of the first aspect of the present invention produces a solvent containing compounds extracted from plant material. As described above, the solvent containing the compounds extracted from the plant material may be mixed with a pesticidally active oil to form a spray formulation for the control of pests. However, the invention is not limited to the use of the solvent containing the extracted compounds for this purpose. In some embodiments of the invention, the solvent is selected such that the solvent containing the compounds extracted from the plant material is suitable for mixing with other components to form surface coating compositions, herbicidal compositions, defoliant compositions, pharmaceutical compositions, cosmetics or foods containing the compounds extracted from the plant material.

The invention is described below by reference to certain non-limiting examples. It will be appreciated by persons skilled in the art that numerous variations and/or modifications may be made to the invention as described in the examples without departing from the spirit or scope of the invention as broadly described. The following examples are, therefore, to be considered in all respects as illustrative and not restrictive.

EXAMPLES

Example 1

The following table shows a comparison between insecticidal efficacy of a solvent containing compounds extracted from plant material prepared by the method of the present invention using the product "Hasten" as the solvent, versus a comparable extract produced using the solvent dimethyl sulphoxide. Dimethyl sulphoxide is a solvent which may be used in conventional solvent extractions of plant materials and is regarded as a powerful solvent.

In each case, dried leaves of *Tasmannia stipitata* from the same bulk sample were used. The same extraction process was carried out for each solvent. The solvent was placed in a vessel equipped with a high shear mixer. Agitation of the solvent was commenced and the plant material added progressively. Agitation was continued until the plant material was dispersed in the solvent. Agitation was then stopped and the mixture allowed to stand at room temperature for 24 hours. The plant material was then separated from the solvent by filtration.

The solvent containing the compounds extracted from the plant material was mixed with water at the percentage by volume listed in the table below (CONC%), and the mixture sprayed on a surface containing Two Spotted Mites and the mortality, feeding and egglaying of the mites was observed. The results are reported in the table below.

| PRODUCT | CONC (%) * | MEAN TWO SPOTTED MITE (TSM) MORTALITY 24h (%) | COMMENTS |
|---|---|---|---|
| *Tasmannia stipitata* extracted with esterfied vegetable oil and surfactants | | | Formed emulsion |
| | 0.5 | 62.7 | No TSM eggs, no feeding |
| | 1.0 | 98.4 | TSM convulsing |
| | 2.0 | 100 | Some phytoxicity |
| | 4.0 | 100 | High phytoxicity |
| | 8.0 | 100 | High phytoxicity |
| *Tasmannia stipitata* extracted with dimethyl sulphoxide | | | Formed clear solution |
| | 0.5 | 3.9 | Normal TSM feeding and egglaying |
| | 1.0 | 2.8 | Some TSM eggs, convulsing |
| | 2.0 | 66.2 | No TSM eggs, no feeding |
| | 4.0 | 92.9 | TSM convulsing, some phytoxicity |
| | 8.0 | 100 | Phytoxicity |

*CONC (%) refers to the percentage by volume of the total extract (i.e. the compounds extracted from the plant material and the solvent) dispersed in water.

This example demonstrates that the solvent containing the extracted compounds produced by the method of the present invention had pesticidal activity against Two Spotted Mites.

Example 2

The method for extracting compounds from plant material described in Example 1 was repeated using the leaves of *Tasmannia stipitata* and the product "Hasten" as the solvent, to produce a solvent containing compounds extracted from the leaves of *Tasmannia stipitata*. The solvent containing the extracted compounds was in the form of a dilute dark green solution. The solvent containing the extracted compounds was combined 50% w/w with a C24 paraffinic spray oil to produce a clear, greenish coloured formulation. This formulation can be used as a spray formulation.

Compounds were also extracted from leaves of *Tasmannia stipitata* using chlorodifluoromethane as the solvent. The solvent was removed by evaporation at or below room temperature, leaving a residue in the form of a resinous paste. The paste would not dissolve into a paraffinic mineral oil, and the addition of various solvents and surfactants to the mixture in an attempt to obtain a stable mixture with the paraffinic oil was unsuccessful.

P/00/001
Section 29

AUSTRALIA
Patents Act 1990

# PATENT REQUEST : PROVISIONAL APPLICATION

I/We, being the person(s) identified below as the Applicant(s), request the grant of a patent for an invention described in the accompanying provisional specification.

| | |
|---|---|
| **Applicant(s):** | NATIVE FIRE PTY LIMITED<br>A.C.N 069 372 167 |
| **Address:** | 3 TERRELL AVENUE<br>WAHROONGA NSW 2076<br>AUSTRALIA |
| **Invention Title:** | EXTRACTION PROCESS |
| **Name(s) of Actual Inventor(s):** | Clive ERSKINE |
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Our Ref: AJ:SDS:GHB:P49460

DATED: 24 October 2003

NATIVE FIRE PTY LIMITED

Patent Attorney for and
on behalf of the Applicant

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. A method for extracting compounds from plant material, the method comprising the step of contacting the plant material with a solvent, wherein the solvent is selected from the group consisting of chemically modified oils, surfactants, mixtures thereof, and mixtures of one or more chemically modified oils and/or one or more surfactants with an oil that is not a chemically modified oil, under conditions effective to extract compounds from the plant material into the solvent.

2. The method as claimed in claim 1 wherein the solvent is selected from the group consisting of chemically modified oils, surfactants and mixtures thereof.

3. The method as claimed in claim 1 or claim 2, wherein the chemically modified oil is an esterified oil.

4. The method as claimed in any one of claims 1 to 3, wherein the chemically modified oil is an esterified animal or vegetable oil.

5. The method as claimed in any one of claims 1 to 4, wherein the chemically modified oil is a methyl, ethyl, propyl or butyl ester of the oil.

6. The method as claimed in any one of claims 1 to 5, wherein the chemically modified oil is an esterified vegetable oil produced from an oil obtained from soybean, sunflower, safflower, canola, cotton, coconut, castor or olive.

7. The method as claimed in any one of claims 1 to 6, wherein the surfactant is a non-ionic surfactant.

8. The method as claimed in any one of claims 1 to 7, wherein the surfactant is selected from the group consisting of glycerol esters of fatty acids; polyethylene glycol dioleate; 9-octadecenoic acid monoester with 1,2-propanediol; polyethylene glycol, dioleate; polyethylene glycol, monococonut ester; polyethylene glycol, monooleate; and diethylene glycol, monooleate.

9. The method as claimed in any one of claims 1 to 8, wherein the solvent is a mixture of about 70% to about 90% by weight of one or more chemically modified oil(s) that is not a surfactant, and about 10% to about 30% by weight of one or more surfactant(s).

10. The method as claimed in any one of claims 1 to 9, wherein the solvent is mixed with one or more second solvents selected from the group consisting of ethanol, acetone, glycerol and hexane.

11. The method as claimed in claim 10, wherein the second solvents comprise about 5% to about 50% by weight of the total solvents used.

12. The method of any one of claims 1 to 11, wherein the plant material is plant material from *Tasmannia stipitata, Prostanthera incisa* or *Callitris glaucophylla.*

13. A solvent containing compounds extracted from plant material produced by the method of any one of claims 1 to 12.

14. A spray formulation comprising a solvent according to claim 13.

15. A spray formulation according to claim 14, further comprising a pesticidally active oil.

16. A method for producing a spray formulation containing compounds extracted from plant material, the method comprising the steps of:

(a) contacting plant material with a solvent, wherein the solvent is selected from the group consisting of chemically modified oils, surfactants, mixtures thereof, and mixtures of one or more chemically modified oils and/or one or more surfactants with an oil that is not a chemically modified oil, under conditions effective to extract compounds from the plant material into the solvent;

(b) separating the solvent from the plant material; and

(c) mixing the solvent with a pesticidally active oil to produce a spray

formulation containing compounds extracted from the plant material.

17. A spray formulation produced by the method of claim 16.

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